



(19)

Europäisches Patentamt
European Patent Office
Office européen des brevets



(11)

EP 0 854 136 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention
of the grant of the patent:
14.11.2001 Bulletin 2001/46

(51) Int Cl 7 C07C 303/16

(21) Application number: 98100905.3

(22) Date of filing 20.01.1998

(54) Process for the production of pure alkanesulfonic acids

Verfahren zur Herstellung reiner Alkansulfonsäure

Procédé de préparation d'acides alkanesulfoniques purs

(84) Designated Contracting States
DE FR GB

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(30) Priority: 20.01.1997 JP 717797

(43) Date of publication of application:
22.07.1998 Bulletin 1998/30

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(56) References cited:

EP-A-0445365

EEF 0854 13

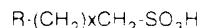
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Description

[0001] This invention relates to a process for the production of an alkylsulfonic acid by the reaction of hydrogen peroxide with an alkylmercaptan, and more particularly it is concerned with a process for the production of an alkanesulfonic acid with high purity in much higher yield and at lower cost.

BACKGROUND OF THE INVENTION

[0002] An alkanesulfonic acid having the general formula



wherein R is a hydrogen atom or the group -OH and x is an integer of $0 \leq x \leq 8$ is publicly known and has been applied for various uses. In particular, a hydroxyalkanesulfonic acid which is encompassed within the above general formula, inter alia, 2-hydroxyethanesulfonic acid also referred to as isethionic acid has a wide variety of utilities. For example, its ester with acrylic acid or methacrylic acid has been used as a reactive emulsifying agent, while its homopolymers or copolymers with vinyl monomers have been employed as a flocculating agent, a dispersing agent, a thickening agent and a flame retardant and the like. Also, its ester with a long chain alkylcarboxylic acid can have an excellent activity as a surfactant and has been widely employed in the field of detergents or cosmetics. Moreover, it has recently been expected typically as bonding agents for basic dyestuffs, adhesives, additives for tin- or tin solder-plating electrolytic bath, and its usage has been widely spread.

[0003] There have hitherto been proposed a variety of processes for the preparation of an alkanesulfonic acid, typically 2-hydroxyethanesulfonic acid(isethionic acid).

[0004] There has been proposed, for example, by Baumstark, et al. in Chem. Ber., 1867, p. 586 a process wherein ethylene is treated with a sulfonating agent such as chlorosulfonic acid or the like or a process wherein ethylene oxide is treated with gaseous sulfuric acid.

[0005] However, it is found that the alkanesulfonic acids produced by these processes are contaminated with unfavorable impurities such as organic chlorinated products, sulfuric acid, organic sulfates or the like and the products are not suited for various application fields such as surfactants, additives for polymers and the like.

[0006] In view of this, many studies have been made for the production of an alkanesulfonic acid having a lower content of impurities.

[0007] For example, Koenig et al. in U.S. Patent No. 2,892,852 have proposed a process wherein an organic thioether or a thioacetic acid ester is reacted with peracetic acid in an acetic acid solution to produce the corresponding sulfonic acid, while there has been proposed a process wherein ozone or a permanganate salt is used as an oxidizing agent in Journal Praktische Chemie, (4), (2), Vol. 27, (1955), pp. 241-242. However, such processes have the problems of oxidizing agents being expensive or an obtainable yield being low so that they have not substantially been utilized in an industrial scale.

[0008] And, U.S. Patent No. 4,499,328 to Longley et al. or Japanese Patent Kokai Application No. 32049-1990 by Iton et al. disclosed a process wherein an alkali salt of isethionic acid with a high yield, which is obtained by reacting ethylene oxide with an alkali salt of bisulfuric acid according to a well-known procedure, is contacted with anhydrous hydrogen chloride in a water-miscible solvent such as an alcohol or the like. However, this process has the drawbacks that a corrosive gas is to be used and also a troublesome procedure for removing by-products of inorganic salts is to be adopted so that it is not advantageous for industrial application.

[0009] Further, U.S. Patent No. 2,727,920 disclosed a process wherein methylmercaptan is subjected to electrolytic oxidation or oxidation with nitric acid. However, low yield is provided in the former case, while accelerated by raising reaction temperature should be controlled in the latter case and both are unsuited for industrial application.

[0010] On the other hand, hydrogen peroxide has favorable conditions such as inexpensive availability, safe handling and by-production of only water after completion of the reaction and the like as compared with other oxidizing agents and there have been made various studies to utilize it for the sulfonation reaction of mercaptan, besides the aforementioned processes.

[0011] However, in the conventional methods, there has been a problem that the yield is low and the purity is not high. Therefore, the present inventors have conducted extensive researches to find out a new method for the production of an alkanesulfonic acid with high purity in much higher yield and at lower cost.

is highly disadvantageous in an industrial scale to carry out the oxidation with a peracid *in situ* as described above [0013]. In the French Patent No. 2,616,786, Deschrijver et al. proposed a process for the production of an alkanesulfonic acid by oxidation of an alkylmercaptan with hydrogen peroxide using a molybdenum or tungsten derivative as a catalyst. However, this process presented the problem of the product being colored and further resulted in prevention of the product from utilization in other application field, owing to the coexisting catalyst.

[0014] McGee et al. proposed in U.S. Patent Nos. 4,910,330 and 4,987,250 a process for the production of an alkanesulfonic acid by oxidation of an alkylmercaptan with hydrogen peroxide without the aforementioned problems such as contamination of an organic acid or a catalyst.

[0015] The process by McGee et al. is excellent in that an alkanesulfonic acid can be produced without any contamination of the product by undesirable additives, but there may be required troublesome steps wherein conc. hydrogen peroxide is diluted with water (for example, up to about 30% by weight), and subsequently the dilution water (and the water by-produced during the reaction) is distilled off to conduct a temperature control in order to remove violent reaction heats. Further, in contacting raw materials with an oxidizing agent, an alkylmercaptan and hydrogen peroxide should be simultaneously fed to a portion of the hydrogen peroxide required for the reaction with exact analysis to effect flow control so as to always provide an excess of H_2O_2 in the extremely limited range more approximate to a stoichiometric amount to the raw material, an alkylmercaptan, and then the reaction steps become complicated, which leads to the drawback that both yield and purity of the so-obtained product may be variable.

[0016] Moreover, oxidation is carried out at a reaction temperature substantially beyond 60°C to afford many undesired by-products of impurities such as sulfuric acid or an organic acid. Furthermore, although a theoretically slightly excess amount of hydrogen peroxide to an alkylmercaptan is used, the amount of the hydrogen peroxide to be used is actually insufficient to complete oxidation of an alkylmercaptan under the aforementioned conditions, whereby, as a matter of fact, the reaction intermediates such as disulfides or the like may remain and the product may be contaminated.

[0017] Accordingly, the product prepared by this process is insufficient in product purity to be applied for the general use of an alkanesulfonic acid, particularly hydroxyethanesulfonic acid.

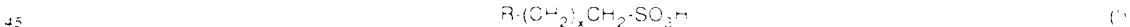
[0018] As explained above, the processes as described in U.S. Patent Nos. 4,910,330 and 4,987,250 involve the serious problems as depicted above for the production using a scale-up or large equipment and thus they are not perfectly satisfactory in an industrial aspect.

[0019] EP-A-0 313 939 discloses a process for preparing alkanesulfonic acids and alkanesulfonyl chlorides which comprises contacting an alkanethiol, a dialkyldisulfide or an alkyl alkanethiol sulfonate mixed in aqueous hydrochloric acid with hydrogen peroxide to produce the corresponding alkanesulfonic acid or alkanesulfonyl chloride showing a low content of undesirable side products arising from side chain chlorination of the alkyl group.

SUMMARY OF THE INVENTION

[0020] We have made studies to solve the problems with the prior art processes relating to a process which comprises reacting an alkylmercaptan with hydrogen peroxide, and as a result found out a process wherein an alkanesulfonic acid of a high utilization value can be produced with high purity and high yield without any need for complex procedures by eliminating the production of impurities which may contaminate the product, and this invention has been completed upon this finding.

[0021] More specifically, this invention relates to a process for the production of an alkanesulfonic acid having the general formula (I)



(wherein R is a hydrogen atom or a group of -OH and x is an integer of 0 to 2) by oxidizing the mercapto group of an alkylmercaptan having the general formula (II)

to distillation with boiling and then said reaction mixture is contacted with an anion exchanger to produce a pure alkanesulfonic acid

[0022] According to the process of this invention, there can be produced the desired alkanesulfonic acid at low cost in high yield and with high purity

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DETAILED DESCRIPTION OF THE INVENTION

[0023] The hydrogen peroxide which may be used in this invention may be any of commercially available products, and its concentration should be not less than 50% by weight, and an aqueous solution thereof of usually not more than 90% by weight, preferably 55 to 65% by weight may be used, whereby the energy cost can be minimized for a concentration step to increase the concentration of the final product of an alkanesulfonic acid up to an extremely high level, and much higher yield of the product can be accomplished

[0024] Specific examples of the alkylmercaptan represented by the general formula (II) may include single compounds such as methylmercaptan, ethylmercaptan, propylmercaptan, butylmercaptan, hydroxymethylmercaptan, 2-mercaptoethanol, 3-mercaptopropanol, 4-mercaptopbutanol and the like and a mixture thereof. The oxidized product obtained by using said alkylmercaptans may be represented by the general formula (I), including the corresponding methanesulfonic acid, ethanesulfonic acid, propanesulfonic acid, butanesulfonic acid, hydroxymethanesulfonic acid, 2-hydroxyethanesulfonic acid(isethionic acid), 3-hydroxypropanesulfonic acid, 4-hydroxybutanesulfonic acid and the like.

[0025] The process of this invention can provide the most superior results particularly when said alkylmercaptan is 2-mercaptoethanol and its oxidized product is 2-hydroxyethylsulfonic acid(isethionic acid). This non-limiting embodiment will be explained in detail hereafter.

[0026] The reaction of 2-mercaptoethanol with hydrogen peroxide may be carried out under atmospheric pressure at a temperature ranging not higher than 50°C, usually 10 to 50°C, and the reaction is desirably carried out at 30 to 50°C, in particular, 40 to 45°C, whereby the production of impurities by-produced by the oxidative decomposition with H₂O₂ such as sulfuric acid, a lower aliphatic acid or the like may be prevented

[0027] In reacting 2-mercaptoethanol with hydrogen peroxide 2-mercaptoethanol and hydrogen peroxide are required usually in a stoichiometric amount on a genuine basis, that is, at 3 moles of hydrogen peroxide per mole of 2-mercaptoethanol, but it is significant to use an excess amount of hydrogen peroxide in order to prevent undesired reaction intermediates from remaining. In this invention, hydrogen peroxide can be used in the range of not less than 3.10 moles up to not more than 40 moles, preferably 3.10 to 3.5 moles, per 1 mole of 2-mercaptoethanol. It is more desirable from the standpoint of a production cost to use hydrogen peroxide in the range of 3.15 to 3.20 moles

[0028] According to the preferred embodiment of this invention, the whole of hydrogen peroxide can be previously charged into the reaction solution and then 2-mercaptoethanol can be fed in a constant amount thereto with a proper stirring. The flow rate in the feeding at that time may be in the range sufficient to maintain a prescribed reaction temperature in compliance with cooling capacity of the reaction vessel, and it is preferable to add the 2-mercaptoethanol illustratively over 3 to 10 hours, desirably 4 to 8 hours, particularly about 6 hours, more or less, whereby complicated procedures may be avoided and a steady temperature control may be effected

[0029] In the process of this invention, it is essential to provide an aging period after completion of the addition of 2-mercaptoethanol. The aging period may be usually 3 to 20 hours, desirably 6 to 10 hours, particularly preferably about 8 hours, more or less, whereby the production of undesired impurities capable of contaminating the product can be prevented. Aging temperature may be desirably in the range that could not induce any side reactions or any excessive oxidation of the isethionic acid as produced, in particular, a temperature of 20 to 60°C which is within the temperature range in feeding 2-mercaptoethanol, in view of easiness of procedures and cost

[0030] At this stage, 90 to 95% of the initial charge amount of hydrogen peroxide is consumed, while there can be confirmed the production of isethionic acid at 90 to 95%, sulfuric acid at 2 to 6%, low molecular compounds such as acetic acid or acetaldehyde at 1 to 5% and intermediates not yet converted to isethionic acid (for example, partial oxidized products of disulfide derivatives) at 3 to 8%

[0031] The reaction mixture after completion of the aging is then subjected to distillation treatment with boiling. This distillation can be carried out by heating at a temperature to maintain the boiling of the reaction mixture under ultimate pressure (i.e., at a vacuum of 10⁻³ mm Hg)

The following examples illustrate the present invention, but they are not intended to limit the scope of the invention. In these examples, the weight percentages of the respective components are based on the total weight of the reaction mixture.

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[0033] This distillation treatment can offer the advantage that the residual reaction intermediates can be completely converted to isethionic acid in addition to the removal of lower aliphatic acids, to afford a much more improved yield.

[0034] According to the process of this invention, it is essential that an aqueous solution containing a high concentration of isethionic acid obtained after completion of the oxidation reaction is contacted with an anion exchanger especially a weakly basic ion exchanger.

[0035] The aqueous solution obtained according to the oxidation reaction of this invention which has usually an isethionic acid concentration of 40 to 60% by weight and a sulfuric acid concentration of 1.8 to 2.5% by weight can be contacted with a weakly basic ion exchanger to perform a selective adsorption of only those acid contents which could not be removed by flushing distillation, such as sulfuric acid, sulfoacetic acid and the like, thereby accomplishing the purification thereof.

[0036] The weakly basic ion exchangers which may be preferably employed in the process of this invention are those weakly basic ion exchangers having a tertiary amine or polyamine as an ion-exchange group. The tertiary amine as used herein is meant to indicate, for example, a compound represented by the following general formula (III)

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25 wherein Q is an acrylic, styrene or phenol polymer group, and R₁ and R₂ may be the same or different and each represents an alkyl group. As the polyamine, there may be preferably employed any straight or branched amine having the above general formula (III) wherein R₁ is a hydrogen atom and R₂ is an ethylenediamine unit. Any shape of the weakly basic ion exchanger may be utilized if commercially available and those resins with a shape of short fibers, a ground product or beads may be recommended in view of their general-purpose properties.

30 [0037] The ion exchangers which may be suitably employed in this invention may specifically include the following ones shown in terms of their trade names:

Duolite A-561, A-568, A-375, A-368, A-378 and A-7;
35 Diaion WA10, WA11, WA20, WA21 and WA30;
Amberlite IRA-35, IRA-60E, IRA-68, IRA-93ZU and IRA-94S;

and others.

[0038] A passing velocity of a treating liquid SV may generally be in the range of 0.1 to 2, and SV is preferably in the range of 0.2 to 1.0, particularly 0.4 to 0.6, in order to recover the desired isethionic acid at higher purity.

[0039] A method for contacting with a weakly basic ion exchanger is not particularly critical and there may be used any conventional method, for example, a method wherein the reaction mixture containing isethionic acid is passed downflow or upflow through said resin previously packed into a resin column.

[0040] An operation temperature is not particularly critical and the desired product having sufficiently high yield and purity can be obtained at ordinary temperature.

[0041] The product at this stage may be usually applied to every well-known uses, but, if coloration of the isethionic acid solution is observed with deterioration of the resin, the reaction mixture after the above treatment may be then passed through an organic material-absorbing resin, for example, Amberlite XAD-4 to perform decolorization.

[0042] According to this invention, isethionic acid may always be obtained in high yield of 95 to 97% and with high purity of 99% or more.

[0043] The isethionic acid thus produced is inexpensive with high concentration and high purity in view of economic definite production thereof, and may be effectively used for various purposes.

[0045] This invention will be more fully explained by way of the following examples, but the invention is not limited thereto.

to these examples

EXAMPLE 1

- 5 [0046] To a glass reaction vessel equipped with an internal condenser, a stirrer, a condenser with an opening and shutting cock for removal of a solvent, and a liquid inlet tube was added 357g(6.3mo.) of 60% by weight hydrogen peroxide all at once, and then 156g(2.0mol) of 2-mercaptoethanol was continuously fed from the liquid inlet tube at 0.4ml/min. while the reaction mixture was vigorously stirred. During the period of this feeding, a liquid temperature was kept at 45°C by adjusting the volume of the cooling water supplied to the condenser.
- 10 [0047] After completion of 2-mercaptoethanol, stirring was continued at room temperature for 10 hours. At this stage, there was afforded a 43.2% by weight isethionic acid solution in water (Yield 221.8g : 88%). Also, in addition to 1.1% by weight sulfuric acid (Yield 5.6g) and 2.5% by weight hydrogen peroxide (Yield 12.8g), there were contaminated low boiling compounds such as 2-hydroxyethyl disulfide polyoxide, acetaldehyde, acetic acid and the like.
- 15 [0048] The reaction mixture was heated to 110°C under atmospheric pressure with vigorous stirring. At this point, nitrogen gas was blown through via a sample inlet tube to partially remove off steam out of the reaction system.
- [0049] After 5 hours, there was given a 55.0% by weight aqueous solution of isethionic acid (Yield 241.9g : 96.0%). In this conc. aqueous isethionic acid solution thus obtained, there was not confirmed any by-products involved therein other than sulfuric acid at a productivity of 2.8% and sulfonated acetic acid at a productivity of 0.2%.
- 20 [0050] The reaction mixture was passed through a 35mm ø column packed with "Duolite A-561" (manufactured by Sumitomo Chemical Co.,Ltd.) which had been regenerated with an alkaline aqueous solution at SV 0.5 from the column top (downflow).
- [0051] The concentrations of isethionic acid and sulfuric acid were quantitatively analyzed by means of an ion chromatography and a neutralization titration.
- 25 [0052] A 54.0% by weight aqueous isethionic acid solution (Yield 241.6g : 95.9%) has a content of sulfuric acid of 0.1% by weight only (Purity of isethionic acid : 99.9%).

EXAMPLE 2

- 30 [0053] Example 1 was repeated except that the oxidation reaction temperature when 2-mercaptoethanol was added was changed to 25°C.
- [0054] There was obtained a 50.2% by weight aqueous isethionic acid solution (Yield 234.4g : 93.0%). The residual amount of sulfuric acid was not more than 0.1% by weight (Purity of isethionic acid : 99.8%).

COMPARATIVE EXAMPLE 1

- 35 [0055] The reaction was carried out in the same manner as described in Example 1 except that the aging period after completion of the addition of 2-mercaptoethanol was not provided.
- [0056] Yield of isethionic acid was 221.8g (Yield 88.0%). Contamination of low-boiling compounds was not observed but contamination of mono- or poly-oxides such as 2-hydroxyethyl sulfide or 2-hydroxyethyl disulfide was confirmed in the solution afforded after treatment of the resin. The solution was slightly yellowish-brown colored.

COMPARATIVE EXAMPLE 2

- 40 [0057] The reaction was carried out in the same manner as described in Example 1 except that the mole ratio of H₂O₂/2-mercaptoethanol was changed to 3.05/1.0(mole/mole).
- [0058] Yield of isethionic acid was 209.2g (Yield 83.1%). Contamination of impurities such as the reaction intermediate, 2-hydroxyethyl disulfide, and mono- or poly-oxide thereof and the like was confirmed in the resulting solution. The solution was slightly yellowish-brown colored.

45 50 COMPARATIVE EXAMPLE 3

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Claims

1. A process for the production of an alkanesulfonic acid having the general formula (I)

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(wherein R is a hydrogen atom or a -OH-group and x is an integer of $0 \leq x \leq 8$) by oxidizing the mercapto group of an alkylmercaptan having the general formula (II)

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15 (wherein R and x are as defined above) using hydrogen peroxide, characterized in that an aqueous hydrogen peroxide solution in the whole amount is charged into a reaction vessel, said aqueous hydrogen peroxide solution having a H_2O_2 concentration of not less than 50% by weight and having a H_2O_2 amount of not less than 3.10 moles per mole of said alkylmercaptan, said alkylmercaptan is substantially continuously fed to said reaction vessel at a reaction temperature never beyond 50°C and thereafter an aging period is provided and the reaction mixture is subsequently subjected to distillation with boiling and then said reaction mixture is contacted with an anion exchanger to produce a pure alkanesulfonic acid.

- 20
2. A process as claimed in claim 1 wherein said aging after completion of the continuous feeding of said alkylmercaptan is carried out at a temperature ranging from 25°C to 50°C for not less than 6 hours
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3. A process as claimed in claim 1 wherein distillation with boiling after completion of the aging is carried out at a temperature of 100 to 120°C while at least one gas selected from the group of air, steam and nitrogen gas is flushed into said reaction vessel.
- 30
4. A process as claimed in claim 1 wherein said anion exchanger is a weakly basic anion exchange resin which has a tertiary amine or polyamine as ion-exchange group
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5. A process as claimed in claim 1 wherein the reaction mixture is contacted with said anion exchanger and subsequently with an organic material-absorbing resin.
6. A process as claimed in claim 1 wherein said alkylmercaptan is 2-mercaptopropano and said alkanesulfonic acid is 2-hydroxyethylsulfonic acid.

Patentansprüche

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1. Verfahren zur Herstellung einer Alkansulfinsäure mit der allgemeinen Formel (I)

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(worin R ein Wasserstoffatom oder eine -OH-Gruppe ist und x eine ganze Zahl ist und $0 \leq x \leq 8$) durch Oxidieren der Mercaptogruppe eines Alkylmercaptans der allgemeinen Formel (II)

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gemisch mit einem Anionenaustauscher unter Herstellung einer reinen Alkansulfinsäure kontaktiert wird

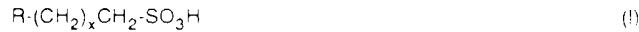
2. Verfahren nach Anspruch 1, wobei die Reaktion nach der vollständigen Durchführung des kontinuierlichen Einspeisens des Alkylmercaptans nicht kürzer als 6 Stunden bei einer Temperatur im Bereich von 25°C bis 50°C durchgeführt wird
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3. Verfahren nach Anspruch 1, wobei die Destillation unter Kochen nach dem vollständigen Ab auf der Reaktion bei einer Temperatur von 100 bis 120°C durchgeführt wird, während mindestens ein Gas, das aus der Gruppe ausgewählt ist, die aus Luft, Dampf und Stickstoffgas besteht, in das Reaktionsgefäß eingeleitet wird
- 10
4. Verfahren nach Anspruch 1, wobei der Anionenaustauscher ein schwach basisches Anionenaustauscherharz ist, das ein tertiäres Amin oder ein Polyamin als Ionenaustauschergruppe hat
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5. Verfahren nach Anspruch 1, wobei das Reaktionsgemisch mit dem Anionenaustauscher und anschließend mit einem Absorptionsharz aus einem organischen Material kontaktiert wird
6. Verfahren nach Anspruch 1, wobei das Alkylmercaptan 2-Mercaptoethanol ist und die Alkansulfinsäure 2-Hydroxyethylsulfinsäure ist.

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Revendications

1. Procédé pour la production d'un acide alcanesulfonique ayant la formule générale (I)

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(dans laquelle R est un atome d'hydrogène ou un groupe -OH et x est un nombre entier de 0 ≤ x ≤ 8) par oxydation du groupe mercapto d'un alkylmercaptan ayant la formule générale (II)

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(dans laquelle R et x sont comme définis ci-dessus) en utilisant du peroxyde d'hydrogène, caractérisé en ce qu'une solution aqueuse de peroxyde d'hydrogène est déposée dans la quantité totale dans une cuve de réaction ladite solution aqueuse de peroxyde d'hydrogène ayant une concentration en H₂O₂ d'au moins 50 % en poids et ayant une quantité de H₂O₂ d'au moins 3.10 moles par mole dudit alkylmercaptan, ledit alkylmercaptan est introduit pratiquement en continu dans ladite cuve de réaction à une température de réaction jamais supérieure à 50°C, et il est après cela fourni une période de vieillissement et le mélange réactionnel est ensuite soumis à une distillation avec ébullition et ledit mélange réactionnel est ensuite mis en contact avec un échangeur d'anions pour produire un acide alcanesulfonique pur

2. Procédé selon la revendication 1, dans lequel ledit vieillissement après l'achèvement de la réaction continue en ledit alkylmercaptan est réalisé à une température comprise entre 25°C et 50°C pendant au moins 6 h
- 45
3. Procédé selon la revendication 1, dans lequel la distillation avec ébullition après l'achèvement du vieillissement est réalisée à une température de 100 à 120°C alors qu'au moins un gaz choisi parmi air, la vapeur d'eau et l'azote gazeux rince ladite cuve de réaction
4. Procédé selon la revendication 1, dans lequel ledit échangeur d'anions est, lorsque non déjà le cas, complètement rempli avec un échangeur d'anions